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NRL Memorandum Report 1660

# Effect of Corrosion Inhibitors on Jet Fuel Filtration

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ABSTRACT

Corrosion inhibitors in JP-4 interact with metals such as iron and zinc to form gelatinous precipitates which give rise to filtration difficulties. The problem is minimal in the absence of free water and is more severe in the presence of sea water than distilled water. Anti-icing additive aggravates the filter plugging phenomenon.

All seven corrosion inhibitors currently approved for use in JP-4 exhibited filter plugging but some were much worse than others.

PROBLEM STATUS

This is a final report on this phase of the problem; work on the problem is continuing.

AUTHORIZATION

NRL Problem C01-01

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# EFFECT OF CORROSION INHIBITORS ON JET FUEL FILTRATION

G. W. McLaren, J. A. Krynitsky, and R. N. Hazlett

## I. INTRODUCTION

In late 1963, a filter separator plugging problem occurred in the fueling system at Ramey AFB in Puerto Rico. The filter plugging tendency was traced to the corrosion inhibitor present in the JP-4 fuel. Local action was taken to eliminate the use of corrosion inhibitor in the fuel used, thereby alleviating the problem.

As a result of this experience, a laboratory study was undertaken to investigate the effect of corrosion inhibitors on the filterability of jet fuels.

Preliminary investigations (1) showed that a number of corrosion inhibitors did, in fact, promote filter plugging tendencies of the fuel when water bottoms were present.

The present study was to evaluate the extent of the problem, to examine the importance of a number of variables, and to determine the interrelationships of various JP-4 fuels with each of the approved corrosion inhibitors.

## II. APPARATUS AND PROCEDURES

A Millipore Silting Index Apparatus (2), modified by the substitution of a 2418 g. drive weight, was used to evaluate filterability. A schematic diagram of the essential parts of the apparatus are shown in Figure 1. Ten ml. samples were withdrawn from the fuel specimen into a glass syringe. These samples were then forced through a filter of 0.8μ pore size and 80% porosity at a constant head of 20 $lb/in^2$. Four different filter areas were used depending on the sample. Filtration times were measured for 1, 5, and 10 ml of fuel filtered by observing the plunger passing bench marks in the syringe.

For each test a value of Silting Index was determined by relating successive times of flow as follows:

$$\text{S. I. (Silting Index)} = \frac{T_3 - 2T_2}{T_1}$$

where $T_3$ = filtration time for 100% of fuel
$T_2$ = filtration time for first 50% of fuel
$T_1$ = filtration time for first 10% of fuel

The filtration area was varied by interchanging filter heads as follows:

#1 head - $1 \times 10^{-4}$ sq. mm.
#2 head $4.3 \times 10^{-4}$ sq. mm.
#3 head $18.5 \times 10^{-4}$ sq. mm.
#4 head $80 \times 10^{-4}$ sq. mm.

If a Silting Index of below 0.5 or above 20.0 was obtained on one head the next lower or higher numbered head was used.

To interpret the data more easily a Reduced Silting Index is used: the silting index was assigned the number of the silting head used, values greater than 10 received a plus following the head number, values greater than 20 were given the value of the next larger silting head.

The filter papers used in most of the work were Millipore AAWP 01300 white, plain filters which have a pore size of 0.8µ ± .05µ. Other papers used were cut from nominal 5 and 10 micron papers.

Water bottoms were synthetic sea water or distilled water. The synthetic sea water was prepared according to ASTM procedure D665-60.

An anti-icing additive was included in some instances. The material used consisted of ethylene glycol monomethyl ether plus 0.4% glycerine.

All seven corrosion inhibitors on the Qualified Products List were evaluated; AFA-1, RP-2, Santolene C, Lubrizol 541, Tolad 244, TRI-182, Unicor M. These were dissolved in toluene and added to JP-4 fuel to give indicated concentrations. The concentration levels were 4 lb/1000 barrels (16 ppm), 20 lb/1000 barrels (80 ppm), and 100 lb/1000 barrels (400 ppm) which are the lower, upper and 5 times upper, concentrations allowed by the specification.

The study was made in two parts. Pilot studies using one fuel were made using a wide range of exposure variables. Then guided by the results, only the more significant variables were selected, and under precisely controlled conditions, three additional fuels were tested. For convenience the tests made with only one fuel are designated Series A and the later experiments as Series B.

The fuel used in Series A was Searsport, Maine Air Force POL Station JP-4. In Series B, Commonwealth Refining Company JP-4, Pure Oil Company JP-4, and Reference Fuel RF-1 (85% Bayol R-34 and 15% Toluene) were tested.

Test Series A

Searsport, Maine JP-4 was prefiltered through a 0.45μ filter and anti-icing additive (5 ml. per liter) and corrosion inhibitor were added. 100 ml. portions of this fuel were placed in glass jars with 20 ml. of water bottoms. Metal test strips (4" x 1" x 1/8") were inserted so that they extended through the fuel and into the water bottoms. Four kinds of metal strips were used: iron, galvanized iron (zinc), aluminum, and magnesium. The samples were allowed to stand quietly and the fuel portions were tested at the end of one week for filterability. Test samples were pipetted from the central portion of the fuel volume.

Test Series B

In Test Series B samples were stored in the dark in undisturbed one quart narrow mouth round bottles. Each sample contained 720 ml of test fuel with corrosion inhibitor added at a concentration of 20lb/1000 barrels. Added water bottoms consisted of 30 ml of synthetic sea water containing 20% anti-icing additive.

Only two types of metal specimens were used in this series. These were 1010 soft-rolled steel and galvanized steel. The test strips were sized 1/4" x 4" x 1/16" to yield a surface/volume ratio of 0.03. Fuel samples were pipetted from the center of the fuel layer in 100 ml aliquots after 3, 7, and 28 days storage at ambient temperatures and tested by the Silting Index Method.

## III. RESULTS AND DISCUSSION

### A. Evaluation of Variables-Series A

A number of factors may be involved in the filtration problem associated with corrosion inhibitors in jet fuel. In preliminary (Series A) studies, many of these variables were explored; namely, (a) the effect of water, (b) the effect of type of metal, (c) the effect of anti-icing additive and (d) the effect of inhibitor concentration.

Studies with Searsport JP-4 containing AFA or Santolene C at 4lb/1000 barrels showed that in the absence of a metal, filtration effects are minimal. Figure 2 indicates that the silting index is about the same in the absence of water, with distilled water bottoms and with synthetic sea water bottoms. When the anti-icing compound was included in the system, a modest increase in silting index was observed.

The addition of a metal to the system increased the silting index. It can be seen in Figure 3 that in the presence of iron, the synthetic sea water/anti-icing additive combination gave the greatest increase. It should be noted that this significant effect was found with the minimum allowable corrosion inhibitor concentration, 4lb/1000 barrels.

Under somewhat different conditions, synthetic sea water and distilled water were compared with respect to the contribution to the problem. In a series including 4 metals and 7 inhibitors in the presence of anti-icing additive, the Silting Index was usually higher when sea water bottoms were used. This set of data which is presented in Tables I (distilled water) and II (sea water) points out the fact that the presence of sea water causes a much more severe filtration problem in many instances. These data were obtained at inhibitor concentrations of 100 lb/1000 barrels, 5 times the allowable limit, to magnify the phenomenon.

There was some variation in the effects of different metals depending on which corrosion inhibitor was used. In general, magnesium exhibited the greatest effect and aluminum the least. Zinc and iron were intermediate in their behavior.

Some Searsport JP-4 fuel was treated by passing it through silica gel to remove polar materials. Testing of treated and untreated fuel in the same manner as Series A showed a slight but not significant improvement in filtration characteristics.

The silting index value was not in direct proportion to inhibitor concentration. From the data it appeared that a sufficient amount of inhibitor caused the phenomenon and addition of larger amounts did not materially increase it. This is shown in Table III.

B. Series B

The data in Series A indicated that the interaction of corrosion inhibitors with four metals of construction can cause filtration problems. Sea water must also be present and anti-icing additive intensifies the difficulty. This information in conjunction with that from other laboratories (1) indicated the need for a more extensive program involving a cooperative study. Three participants, U. S. Naval Research Laboratory, Aeronautical Engine Laboratory at Philadelphia, Pennsylvania, and Aero Propulsion Laboratory at Wright-Patterson AFB arranged a program involving the variables referred to in the Apparatus and Procedures Section, Series B.

The results of the cooperative study have been summarized by the Aero Propulsion Lab (4). In general, the agreement between laboratories was good and the Silting Index Apparatus was found to be a useful tool for working with filtration problems.

Recent evaluation of the data generated in these studies has led to revision of JP-4 fuel procurement so that a corrosion inhibitor cannot be added without prior approval of the procuring agency (5).

The NRL data for the 7-day storage period are presented in Figures 4 and 5. To be noted are the facts (a) that the metals must be present to effect a filtration problem, (b) that the type of fuel is of minor importance, (c) that zinc and iron behaved similarly with most fuel/inhibitor combinations, and (d) that each inhibitor exhibited a significant increase in Silting Index with at least one fuel.

Particle formation began in a matter of hours and in general the Silting Index was at a maximum from about three days to a week. After several weeks the Silting Index dropped due possibly to settling out, particle growth or agglomeration. Typical data are shown in Figure 6.

The specific corrosion inhibitor used appears to be the most significant variable in this study. In overall behavior, TRI-182 gave the least filter plugging increase with Unicor M next in line. Tolad 244 and Lubrizol 541 gave the greatest silting index increase.

C. Properties of Particulate Matter

The material that plugged the filters was a gelatinous precipitate. The amount present was determined on two selected samples by filtering and weighing residues on HAWP 04700 Millipore filters. These filters are 47 mm. in diameter and have a pore size of 0.45μ.

One liter fuel samples were aged for three days and the particulate matter collected. Pure Oil Company JP-4 was exposed to iron in the presence of synthetic sea water containing 20% by volume anti-icing additive. AFA and Lubrizol 541 inhibitors (20 lb/1000 barrels) afforded 3.3 and 3.2 mg/liter of particulate matter respectively when compared with control samples.

An attempt was made to correlate the turbidity of fuel samples with Silting Index. The turbidity of 46 samples was measured as percent light transmittance at 520 $m\mu$ in a photoelectric colorimeter. The percent transmittance values were compared with Silting Index values. The data were quite scattered and no correlation could be made.

A few experiments were carried out with $5\mu$ and $10\mu$ pore size filters for comparison with the $0.8\mu$ filters used in the Silting Index test.

A Pure Oil JP-4 sample containing 20 lb/1000 barrels of Lubrizol was tested after two months storage over sea water containing 20% AIA in the presence of zinc. The Silting Index for $0.8\mu$ paper was 34.1 on a #2 filter head and for $5\mu$ paper it was -0.25 on #1 filter head.

After swirling the sample to bring up fuel immiscible particles that might have settled to the interface, the Silting Indices were determined again. The standard test using $0.8\mu$ paper gave a Silting Index of 5.5 on the #4 head, $5\mu$ paper gave 19.6 on the #1 head and $10\mu$ paper gave 6.1 on the #1 head. Other tests of Pure Oil and Commonwealth JP-4 with 20 lbs/1000 barrels of corrosion inhibitors stored in the presence of iron and zinc, sea water and anti-icing additive for 10 days, gave zero or negative Silting Indices with $5\mu$ filter paper when the samples were not shaken even though $0.8\mu$ paper gave results in the expected range. However, upon shaking the sample the results were as shown in Table IV.

Since the undisturbed fuel would not plug the $5\mu$ paper when run using the normal volume of a Silting Index test, a larger volume was tried. A four day old sample containing AFA-1 which had $SI_2 = 12.5$ with an $0.8\mu$ filter took approximately 300 ml to plug a $5\mu$ filter in a #1 head.

The data on particle size and filtration through large pore filters are consistent. In the standard test with 0.8μ filter paper, the majority of the particulate matter would be retained and the silting index would be high. With paper pore size larger than the particle size, the filtration would not hold up the particles and the silting index would be low. When larger particles which have settled out with aging are distributed throughout the fuel by swirling the Silting Index with the larger pore size papers exhibits an increase as expected.

## IV. SUMMARY AND CONCLUSIONS

1. All of the seven currently approved materials which are effective as corrosion inhibitors, form a gelatinous precipitate which tends to plug fuel filters under certain conditions. These conditions include the presence of a metal such as iron or zinc and water.

2. Anti-icing additive contributes to particle formation.

3. Particle formation can be obtained with currently used typical JP-4 fuels.

4. These particles tend to settle out in storage systems after several weeks as long as there is no agitation.

5. The Modified Silting Index Apparatus is a useful method for testing the filterability of jet fuels.

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3. Jet fuel, Grades JP-4 and JP-5, MIL-J-5624 F, Sept. 25, 1962.

4. Filterability of Turbine Fuels, Silting Index Apparatus Cooperative Test Program, Aero Propulsion Lab, Wright Patterson AFB, Ohio, Feb. 19, 1965.

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Table I

SERIES A - EFFECT OF WATER AND METALS ON REDUCED SILTING INDEX

| Metal | AFA | SanC | Lubrizol 541 | RP-2 | Tex. TRI182 | Tolad 244 | Unicor M |
|---|---|---|---|---|---|---|---|
| Fe | 1+ | 1 | 1 | 1 | 1 | 2 | 3 |
| Zn | 1 | 1 | 2 | 3 | 1 | 3 | 1 |
| Al | 1 | 1 | 1 | 1 | 1 | 2 | 1 |
| Mg | 3 | 1 | 4 | 3+ | 1 | 3 | 1 |

Distilled water + anti-icing additive
1 week exposure
conc. inhibitor - 100 lb/1000 barrels

Table II

SERIES A - EFFECT OF WATER AND METALS ON REDUCED SILTING INDEX

| Metal | AFA | SanC | Lubrizol 541 | RP-2 | Tex. TRI182 | Tolad 244 | Unicor M |
|---|---|---|---|---|---|---|---|
| Fe | 3+ | 3 | 4 | 3 | 3 | 4 | 3 |
| Zn | 4 | 2 | 4 | 3 | 1 | 4 | 3+ |
| Al | 4 | 1 | 4 | 3 | 1 | 4 | 1 |
| Mg | 4+ | 3 | 4 | 4 | 4 | 4+ | 4 |

Sea water and anti-icing additive
1 week exposure
conc. inhibitor - 100 lb/1000 barrels

Table III

EFFECT OF CONCENTRATION OF INHIBITOR ON REDUCED SILTING INDEX

| | Inhibitor Concentration (lb/1000 barrels) | | |
|---|---|---|---|
| | 4 | 20 | 100 |
| **IRON** | | | |
| AFA | 1 | 4 | 4 |
| San-C | 1 | 3 | 3 |
| **ZINC** | | | |
| AFA | 1 | 2+ | 4 |
| San-C | 2 | 3 | 2 |

Searsport fuel
Sea water and anti-icing additive
One week exposure

Table IV

SILTING INDEX WITH 5 MICRON PAPER

| INHIBITOR | SILTING INDEX #1 Head |
|---|---|
| AFA | + 0.4 |
| RP-2 | 0 |
| San-C | + 1.3 |
| Tolad | + 1.25 |
| Lubrizol | +14 |
| Texaco Tri 182 | + 0.2 |
| Unicor M | + 1.5 |

(Sample stored for 10 days, shaken before measurement)
Commonwealth Ref. Co. JP-4

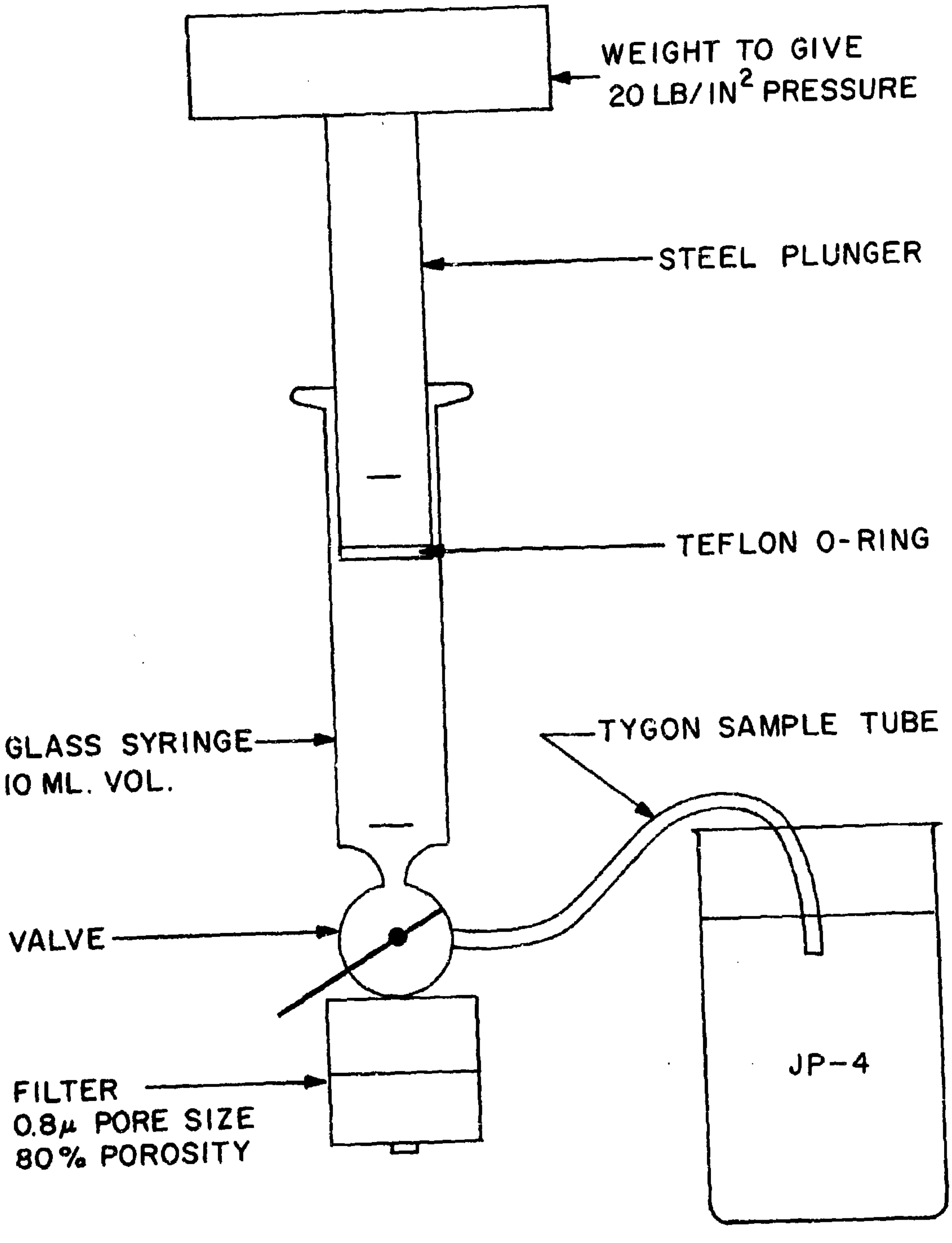


Fig. 1 - Schematic of silting index apparatus

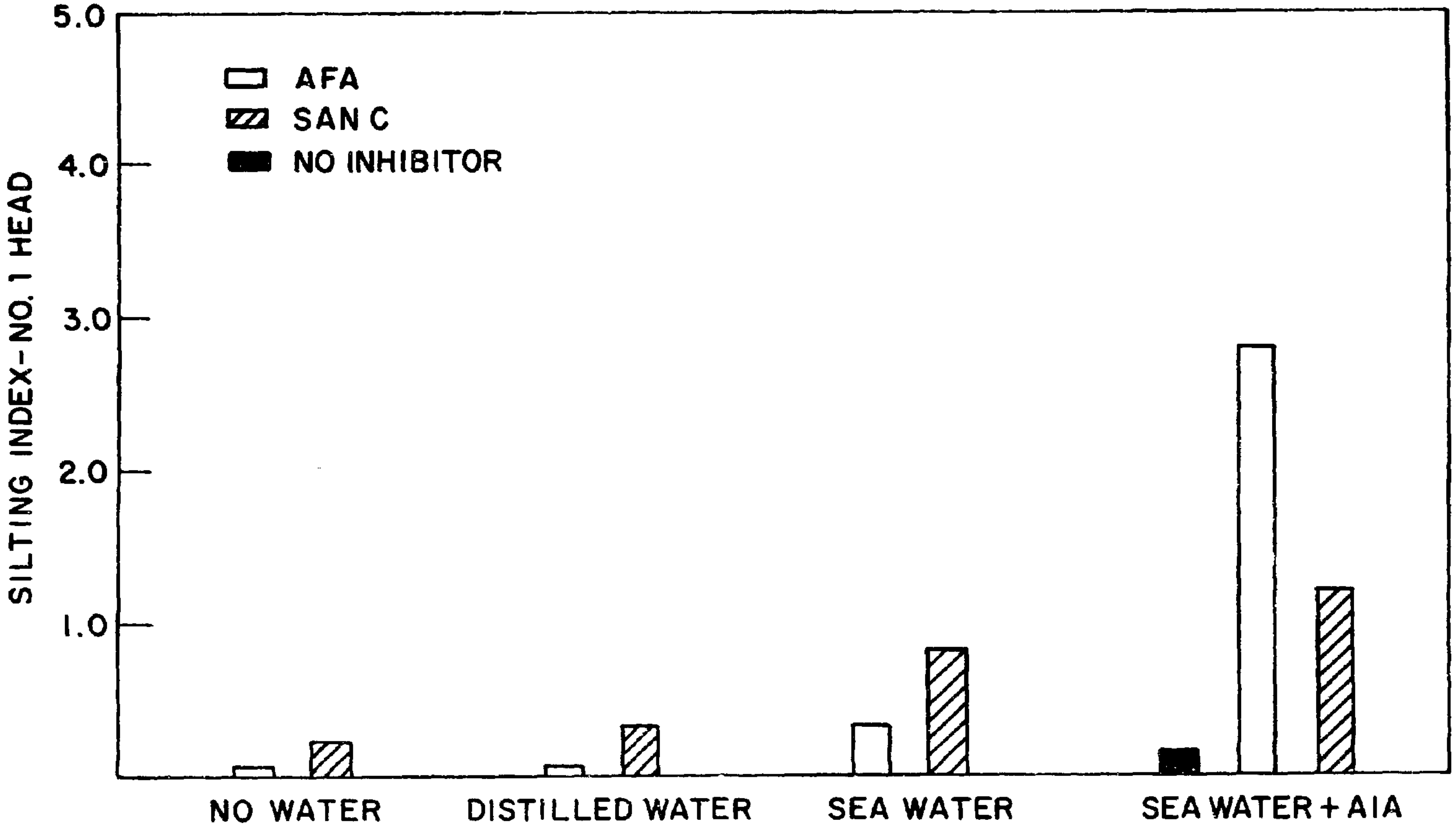


Fig. 2 - Effect of water and anti-icing additive (AIA) on silting index: inhibitor at 4 lb/1000 barrels – no metal strips – 1 week storage

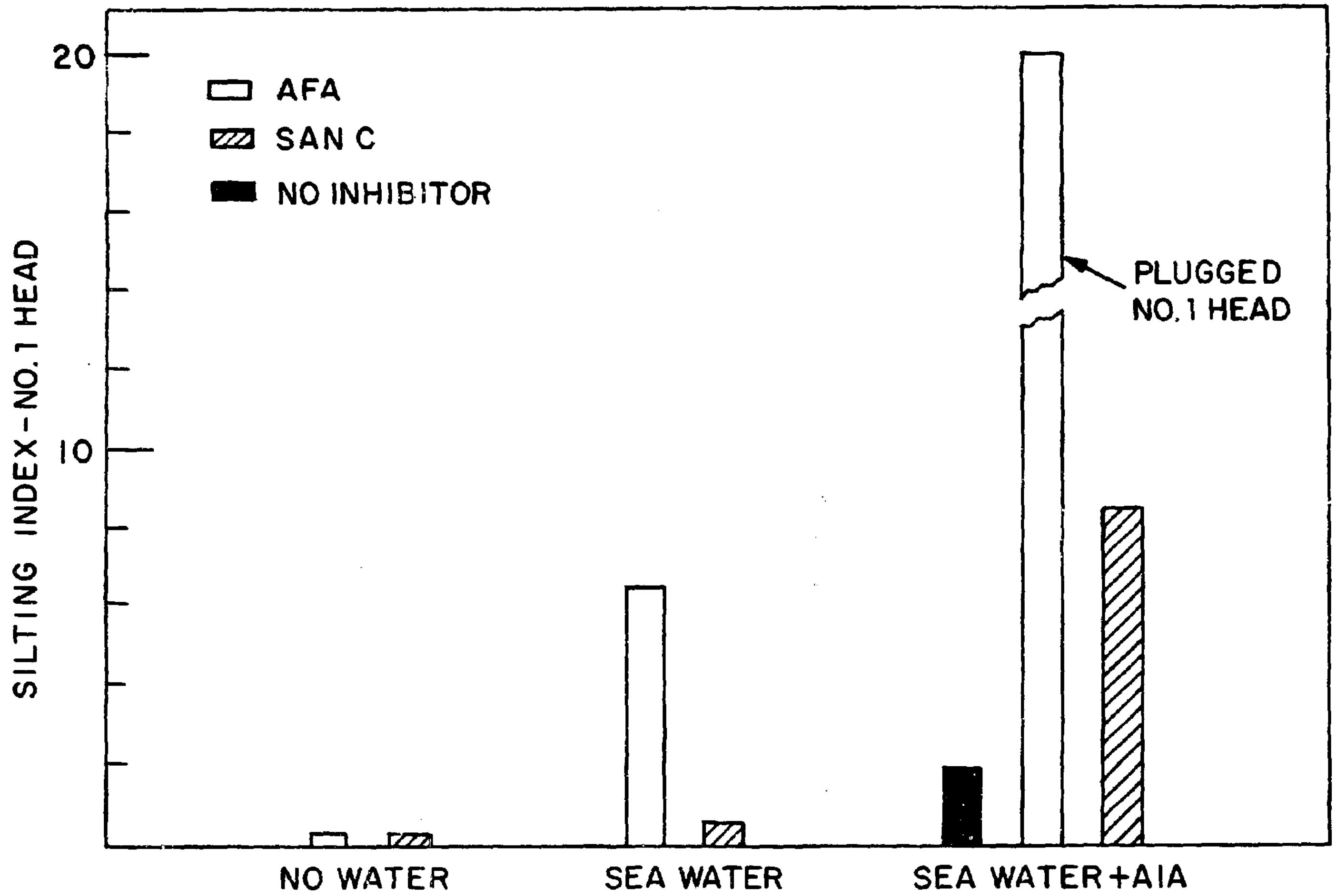


Fig. 3 - Effect of water and anti-icing additive (AIA) on silting index: iron strips - 1 week storage - inhibitor at 4 lb/1000 barrels

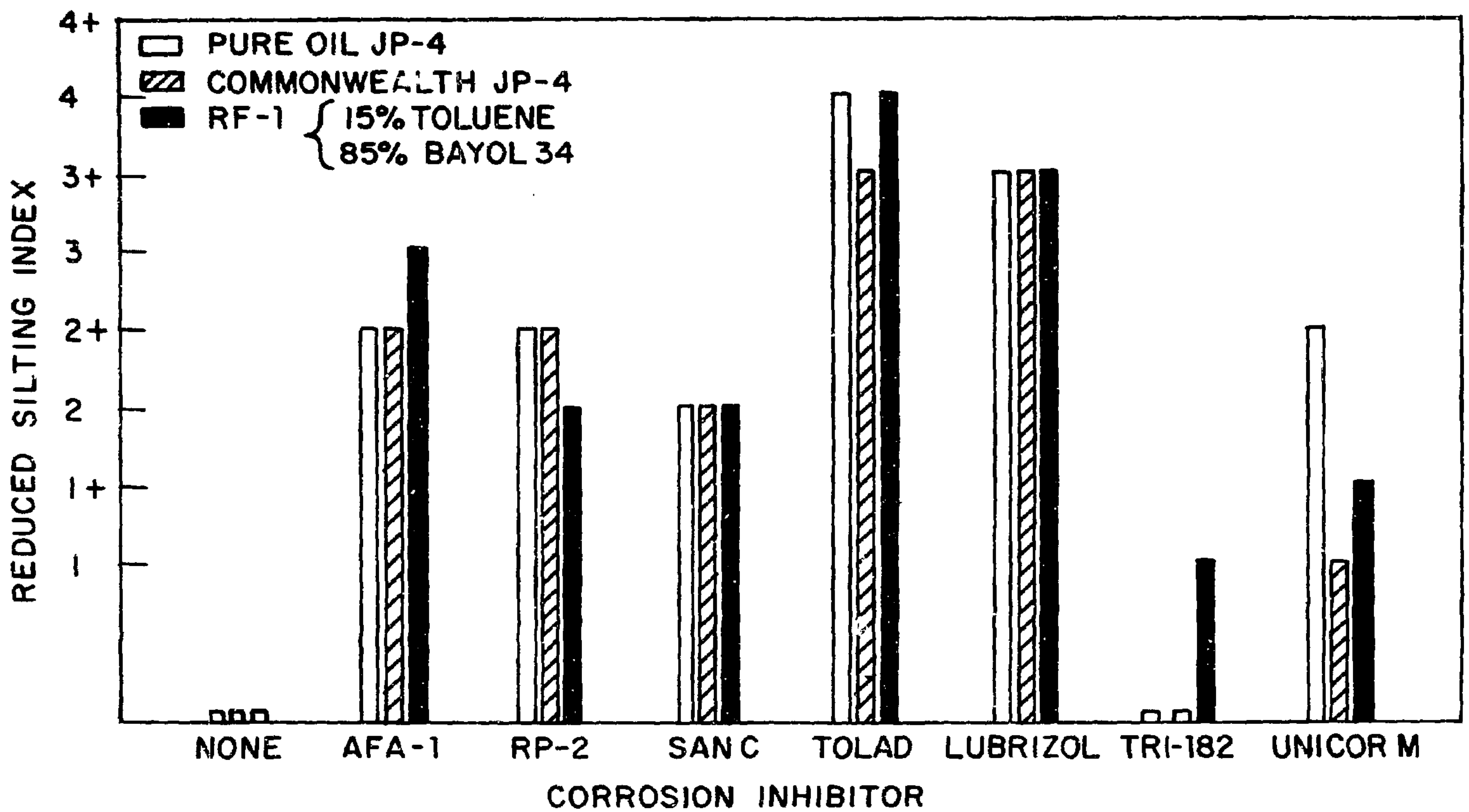


Fig. 4 - Series B: seven day storage for iron strips

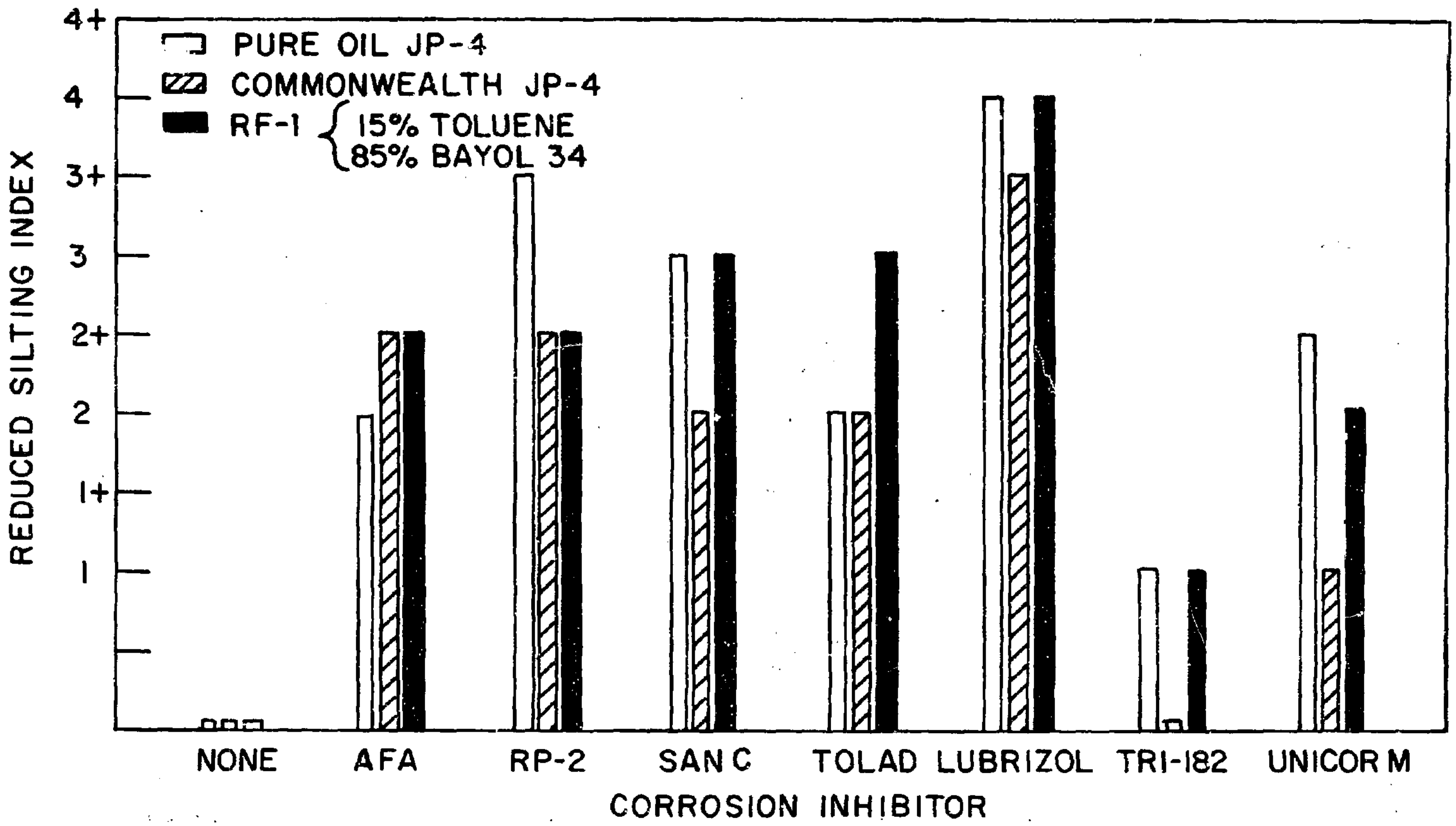


Fig. 5 - Series B: seven day storage for zinc strips

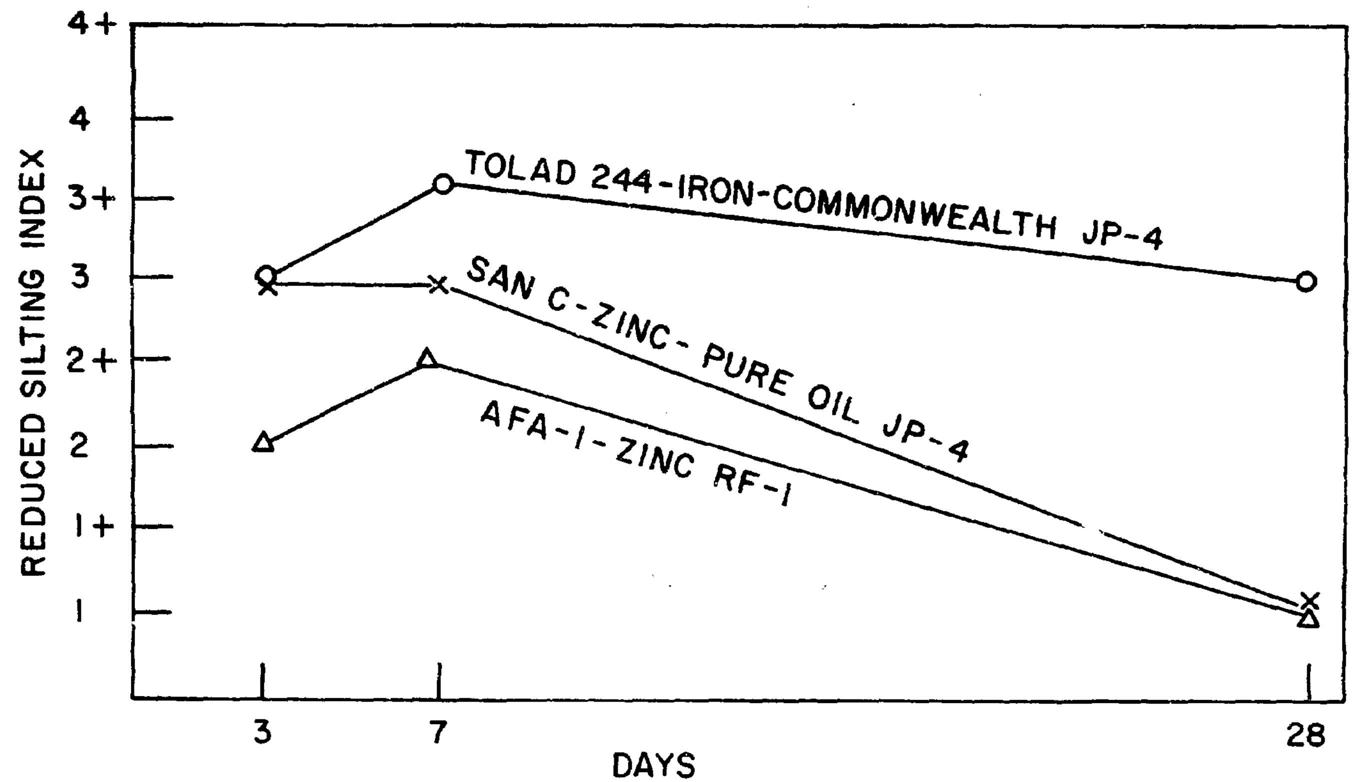


Fig. 6 - Typical change of silting index with time – Series B

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